Dynamic fracture behaviour of some brittle materials

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Abstract

The aim of the present contribution is to study the dynamic fracture properties of brittle materials $MoSi_2$, Si_3N_4 and WC-Co. Dynamic tests at different loading rates (v = 0.992, 1.916, 3.677 m s^{-1}) were carried out using an instrumented Charpy impact pendulum. The force signals were recorded as a function of time by a digital oscilloscope. The stored data were analysed with a spreadsheet procedure. Microstructure analysis was realised by optical microscope and SEM using plasma or chemical etched samples. Fracture surfaces were investigated by SEM. The impact energies for $MoSi_2$ and Si_3N_4 are about 0.02–0.06 J, respectively. The results revealed that the most brittle material is $MoSi_2$ and the systems of WC-Co are the toughest ones. The impact energies of WC-Co systems are between 0.06–1 J, their value depends on the microstructure and increases with increasing volume fraction of Co and grain size of WC grains.

K e y w o r d s: instrumented impact testing, brittle materials, fractography

1. Introduction

High hardness, good wear and oxidation resistance of advanced ceramics are very attractive for both room and high temperature applications. Their wider use as materials for different components is, however, still limited by their low reliability and high price. This is connected with low fracture toughness and with the presence of defects in these materials in the form of pores, clusters of grains, whiskers, inhomogeneities, etc., or with the surface defects, as grinding cracks [1]. Structural ceramics and hard metals, thanks to their high hardness, are often used as cutting tools, which are loaded also in a dynamic way. Therefore, it is very important to know the effect of loading rate on their mechanical and fracture behaviour. The most widely used testing technique for the determination of these properties is the instrumented impact test. There is a lack of investigation concerning the dynamic behaviour of brittle materials as $MoSi_2$, Si_3N_4 and WC-Co as potential candidates for structural application and as widely used materials for cutting tools.

Materials based on $MoSi_2$ are promising candidates for wide variety of elevated temperature structural applications thanks to their high melting point (2030 ℃), excellent oxidation and corrosion resistance, and high temperature ductility above the brittle-ductile transition temperature, in the vicinity of 1000 ℃ [2–4]. However, the main disadvantage, limiting their utilisation, is the low fracture toughness at lower temperatures (< 1000 ℃) and low strength and creep resistance at high temperatures. In order to improve the mentioned properties various approaches based on incorporation of SiC, Nb and ZrO_2 particles, or SiC whiskers into the matrix have been used [5, 6].

Silicon nitride ceramics have been intensively investigated during the last decade with the aim to improve their room and high temperature properties. Using microstructural design (self-reinforcement by large elongated grains and tailoring the chemistry of the intergranular phase) a room tempera-

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ture fracture toughness of more than 10 MPa $m^{1/2}$ and fracture strength higher than 1 GPa have been achieved [1].

Hardmetals based on WC-Co are well known materials for many applications as cutting tools or wire drawing tools. Microstructure of a ductile phase reinforced WC-Co can be fully described by the volume fraction of binder phase – f_{Co}, the mean grain size of carbides – D_{WC}, the mean free path in the binder – L_{Co} and the contiguity of carbide phase – C_{WC} [7]. Number of authors [8] carried out experiments with the aim to find a relationship between the above mentioned microstructure parameters, toughening mechanisms and hardness, strength and fracture toughness.

The aim of the present contribution is to study the room temperature dynamic and fracture behaviour of three brittle materials ($MoSi_2$, Si_3N_4, WC-Co), with different fracture toughness, from approximately 2 MPa $m^{1/2}$ to 20 MPa $m^{1/2}$.

2. Experimental materials and methods

Three brittle materials were used in the investigation: $MoSi_2$, Si_3N_4 and WC-Co with different volume fractions of Co and different grain size of WC. The monolithic $MoSi_2$ was pressureless sintered by Cesiwid, Elektrowarme, GmbH (Erlangen, Germany). The monolithic Si_3N_4 used in this investigation is a gas pressure sintered silicon nitride, prepared using SNE--10 powder with addition of 3 wt.% Y_2O_3 and 1 wt.% Al_2O_3. The WC-Co specimens were prepared using standard preparation technique with three different volume fractions of binder phase and three different grain sizes of WC grains.

Microstructures of the materials were observed on the cut, polished and etched (plasma and/or chemically) samples using optical and scanning electron microscopy.

Specimens for dynamic tests were cut from the sintered plates and their surface was ground by diamond wheels and polished using diamond pastes up to 3 μm. Dimensions of Si_3N_4 specimens were 3 × 4 × 44 mm^3, of $MoSi_2$ 3 × 4 × 50 mm^3 and that of WC-Co 4.5 × 4.5 × 44 mm^3.

The dynamic test was carried out on un-notched samples at room temperature, using an instrumented Charpy impact pendulum of 15 J maximum energy, with three different impact velocities ($v = 0.992, 1.916, 3.677$ m s^{-1}). The force and the electric emission signals were recorded as a function of time by a digital oscilloscope [8]. The stored data were analysed with a spreadsheet procedure. The characteristic values of energy, force and displacement were determined.

Fracture surfaces of the investigated materials after performed Charpy test were studied by SEM using macro- and microfractography [9].

3. Results and discussion

Three different phases were identified in the monolithic $MoSi_2$ by EDAX: $MoSi_2$ grains, amorphous SiO_2 and a low amount of Mo_5Si_3 (the hexagonal Nowotny phase). Mean grain size of the $MoSi_2$ grains is approximately 7 μm. TEM observations proved that silica (SiO_2) particles were frequently present in the triple grain junctions of $MoSi_2$ grains and occasionally they were placed also intragranularly, inside the $MoSi_2$ grains. The only defects present in the material were pores sized up to 25 μm. The Si_3N_4 has a bimodal grain size distribution, with large grains, up to 20 μm in diameter and small ones, < 1 μm in diameter. Microstructure of the WC-Co can be characterized by microstructural parameters as the mean grain size of WC grains D_{WC}, mean free path in the binder phase – L_{Co} and the contiguity – C_{WC}. No defects have been found in the Si_3N_4 and WC-Co systems.

The registered force signals at the velocity of 1.916 m s^{-1} for $MoSi_2$, Si_3N_4 and WC-Co are shown in Fig. 1a–c. All materials behaved as perfectly brittle. In the case of $MoSi_2$ the maximum force was achieved in a very short time after the impact and immediately an unstable crack propagation has occurred. In the case of Si_3N_4 and WC-Co the force increased linearly up to a maximum force, where unstable crack propagation occurred. This indicates that in case of all investigated materials no plastic deformation occurs before fracture formation. Considerable differences in maximum force values were found for different materials. The average level of the maximum force was 0.8 kN in the case of $MoSi_2$ and Si_3N_4 approximately and between 2 and 6 kN in the case of WC-Co. At the impact velocity of 3.677 m s^{-1} the average time period with an increased signal was approximately 0.025 ms in the case of $MoSi_2$, in Si_3N_4 – 0.05 ms, and in WC-Co between 0.1 and 0.2 ms.

The impact energies of Si_3N_4, $MoSi_2$ and WC-Co (with different volume fractions of Co and with the grain size of WC) at three different impact velocities, analysed with a spreadsheet procedure, are illustrated in Fig. 2. The impact energy of all studied materials increased with increasing impact velocity, however the differences for the systems $MoSi_2$ and Si_3N_4 are low. The impact energies for $MoSi_2$ and Si_3N_4 were about 0.02 J and 0.06 J, respectively. The impact energy for WC-Co was between 0.06 J and 1 J, depending on the material microstructure.

The impact energy of WC-Co increased with the volume fraction of Co (Fig. 3) and with the grain size of WC (Fig. 4). Such a behaviour is in a good agreement with results of the investigation of microstructure influence on the fracture strength and toughness of cemented carbides. According to these results the ductile second phase of cobalt is the origin of reinforcing mechanisms leading to an increased strength

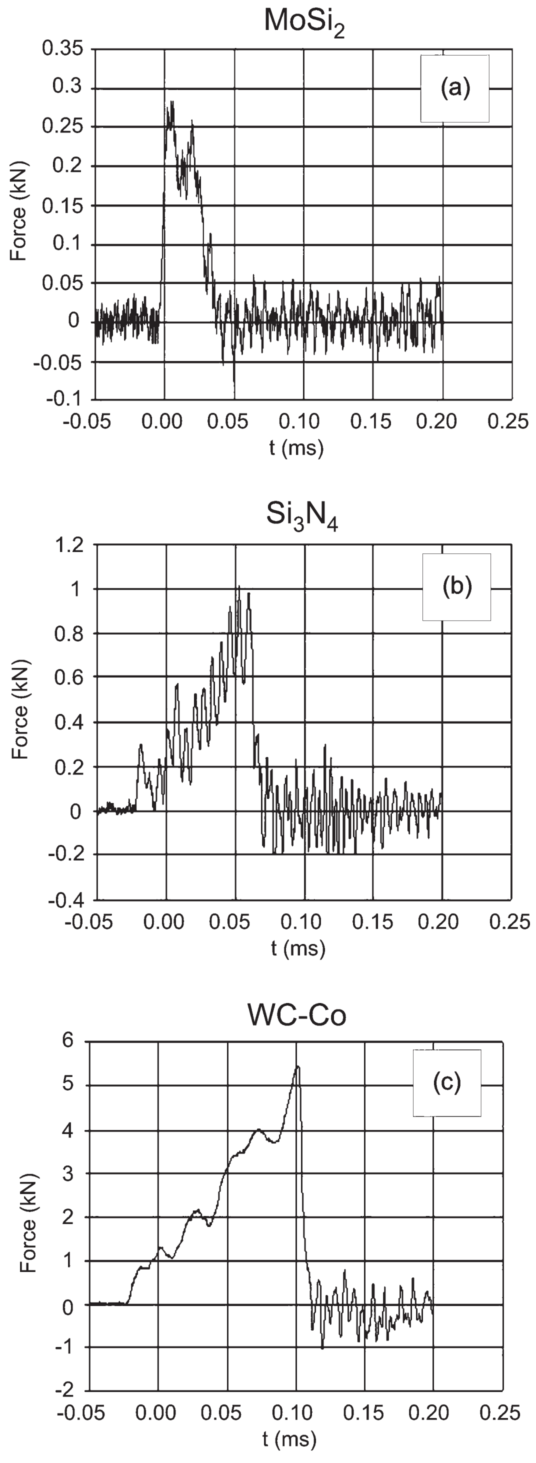

Fig. 1. Force signal for $MoSi_2$ (a), Si_3N_4 (b), and WC-Co (c).

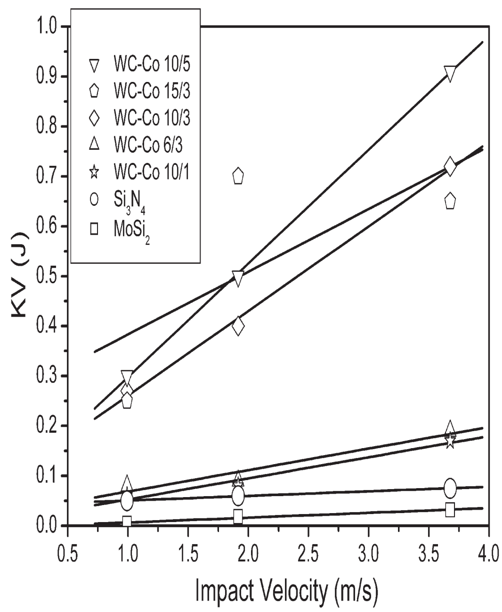

Fig. 2. The impact energy of all studied materials at different impact velocities.

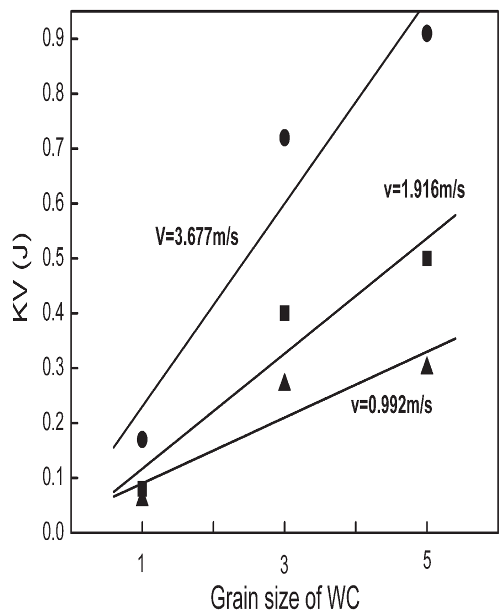

Fig. 3. Effect of the grain size of WC grains on the impact energy of WC-Co materials at different velocities.

and fracture toughness. Frequency of the occurrence of toughening mechanisms increases with increasing the volume fraction of cobalt and increasing the grain size of WC.

The values of impact energy reduced to the specimen dimensions are summarized in Table 1.

Macrofractography of the tested specimens has not revealed fracture origins in the form of technological or surface defects. It seems that in most cases the fractures originate from surface defects and it can be hardly identified. Fracture surfaces of the specimens studied by microfractography are shown in Fig. 5a–c. The $MoSi_2$ exhibits a totally brittle character, mainly with transgranular fracture of $MoSi_2$ grains. The occurrence of intergranular fracture mechanisms is very

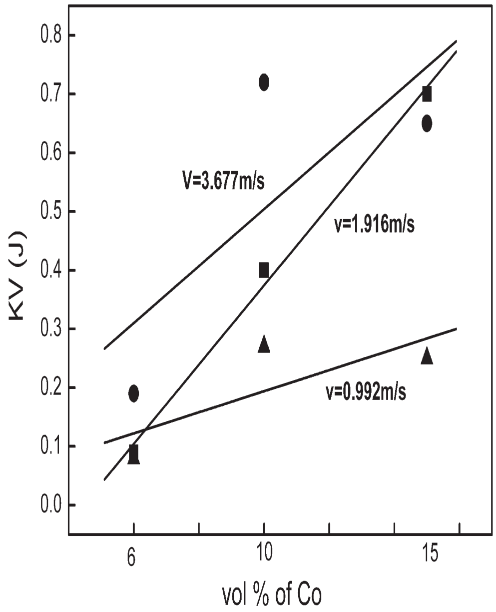

Fig. 4. Effect of the vol.% of Co on the impact energy of WC-Co materials at different velocities.

T a b l e 1. Impact energy of Si_3N_4, $MoSi_2$, and WC-C

Specimen	KC (J cm^{-2})		
	(3.67 m s^{-1})	(1.916 m s^{-1})	(0.992 m s^{-1})
$MoSi_2$	0.26	0.15	0.04
Si_3N_4	0.625	0.5	0.4
WC-Co 10/3	3.55	1.97	1.33
WC-Co 10/5	4.5	2.46	1.46
WC-Co 10/1	0.83	0.39	0.29
WC-Co 15/3	3.2	3.45	1.23
WC-Co 6/3	0.93	0.44	0.39

limited even at the lowest loading rate. The fracture surface of Si_3N_4 is quasi-brittle, a toughening effect of the whiskers like Si_3N_4 grains is evident. However, the potential of toughening mechanism is strongly limited and only in a few cases significant crack deflection or/and whisker pull-out was identified. Toughening in the WC-Co through the binder phase in the form of dimples and necks is more pronounced in materials with higher Co content and higher grain size of WC. Probably there exists a critical value of the microstructural parameter – L_{Co} below which the material is similarly brittle as $MoSi_2$ or Si_3N_4 (see Fig. 2) with a similar impact energy value.

Dynamic fracture behaviour of three brittle materials ($MoSi_2$, Si_3N_4, WC-Co) at room temperature has been investigated. The results revealed that the impact energy values of the studied materials are in good agreement with their fracture toughness and bending strength values. The lowest impact energy was achieved for the most brittle material, $MoSi_2$, and the highest one for the most tough – the WC-Co systems.

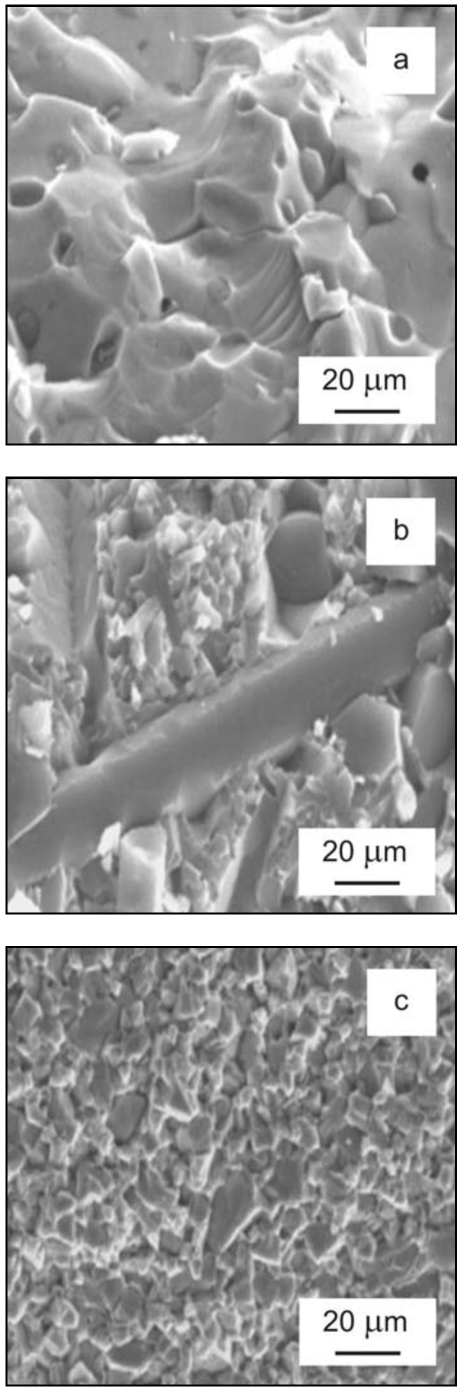

Fig. 5. Fracture surface of the $MoSi_2$ (a), Si_3N_4 (b), and WC-Co (c) after Charpy test.

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